General Disclaimer

One or more of the Following Statements may affect this Document

- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.

- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.

- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.

- This document is paginated as submitted by the original source.

- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.

SUMMARY REPORT

An Experimental Study of the Cryoentrainment Pump and the Behavior of Nude Ionization Gauges at Low Temperature.

J. A. Daggerhart

Department of Mechanical and Aerospace Engineering

North Carolina State University

Raleigh, North Carolina 27607

September 28, 1970

NASA Grant NGR 34-002-106

SUMMARY REPORT

An Experimental Study of the Cryoentrainment Pump and the Behavior of Nude Ionization Gauges at Low Temperature.

J. A. Daggerhart

Department of Mechanical and Aerospace Engineering

North Carolina State University

Raleigh, North Carolina 27607

September 28, 1970

NASA Grant NGR 34-002-106

TABLE OF CONTENTS

INTRODUCTION 1

EXPERIMENTAL PROGRAM 3

DISCUSSION OF RESULTS 7

PLAN FOR FUTURE WORK 13

CONCLUSION 15

INTRODUCTION

To provide a vacuum free of contamination, cryopumping of a system has largely been accepted as a very promising approach. However, even at ultra-low temperature -- $4^{\circ}K$ for example -- helium is not removed from the environment and other techniques must be employed to remove this component.

Based on economic considerations, the desirability of utilizing $4^{\circ}K$ as a condensing temperature is questionable as the cost of refrigeration equipment spirals as the temperature is lowered below $77^{\circ}K$. Thus considerable interest has been shown in using $77^{\circ}K$ as the refrigerant temperature. At this temperature, however, the normal cryopuming mechanism cannot be relied on as the sole pumping action. This restriction is imposed since only carbon dioxide and water vapor, in a system initially air filled, are removed by condensation at $77^{\circ}K$.

To bypass this restriction and still use the $77^{\circ}K$ refrigerant temperature, it has been postulated that a gas stream, easily condensable at this temperature, be injected into the system. This gas stream would then entrain the non-condensable species in the chamber and subsequently trap them in the gas stream condensate formed on the $77^{\circ}K$ surface. Then if the vapor pressure of the pumping gas stream is sufficiently low at $77^{\circ}K$, this entrainment and trapping action can be used as a cryoentrainment pump.

In a preliminary study of the operation of such a device, certain desirable characteristics of the pumping medium may be specified, such as:

(1) an extremely low vapor pressure at $77^{\circ}K$,

(2) low toxic effects,

(3) a high molecular weight to gain entrainment efficiency, and

(4) a condensate impermeable to the non-condensable molecules and with a high thermal conductivity to permit large frost buildups.

Based on these requirements and a desire to easily obtain large quantities of the pumping gas, a commercially available flucocarbon was selected. With a molecular weight of 200 and being available in quantity, octofluorocyclobutane (C_4F_8) was chosen as the primary test pumping gas.

A second study was also begun, during the latter stages of the cryoentrainment pump evaluation, to examine the behavior of ionization gauges operating in a low pressure, low temperature environment. This effort stemmed from observations of the behavior of ionization gauges during the course of an investigation of the thermal transpiration effect.

Theoretical predictions for the thermal transpiration effect in long tubes indicate that the particle number density at the cooled end of the tube (77°K) will be higher than that at the warm end. Thus the indication of an ionization gauge, which measures density, mounted in the cold environment will be higher than that of one mounted at the warm end of the tube.

During the course of the experimental investigation, however, the opposite result was noted. For certain conditions of pressure and temperature, the indication of the cooled ionization gauge was as much as 100 times less than that of the gauge at room temperature.

After eliminating such obvious causes as current leakage and cold wall helium pumping as not being large enough to cause such a distortion of the expected results, an experimental program was begun to at least map, if not explain, the gauge behavior in a cold environment.

EXPERIMENTAL PROGRAM

The basic philosophy behind the design of the cryoentrainment pump was to provide an accelerated jet of the injected pumping gas which would sweep the non-condensable gases from a volume and penetrate a large surface area condenser. To achieve this goal, the experimental apparatus was constructed as indicated in Figure 1.

Two entirely separate structures, fabricated from stainless steel, were employed to provide the actual pumping volume and an outer shielding chamber. The outer shielding is in the form of a bell jar 1.32m in diameter and 1.73m high.

The pumping system, provided for the initial evacuation of both chambers, consists of a 10 and 16 inch oil diffusion pump in parallel with the system and backed by a 50 cfm mechanical pump. The requirement of two oil diffusion pumps to reach base presures near 10^{-6} torr was imposed by the partial blockage of one pump inlet by the inner structure and the large outgassing load generated by various internal fixtures.

The inner structure, containing a 900 liter pumping volume, is initially conical in shape to match the 15^{o} half angle of the nozzle used to accelerate the pumping gas from the stagnation chamber. The lower section of the pumping volume returns to a cylindrical shape to hold the condenser array.

The condenser array is formed form copper tubing wound into the form of concentric cylinders 91.5 cm in height. Four cylinders are used with diameters of 20.3, 40.6, 60.0, and 81.2 cm to give a total condensing surface area of 11.62 sq. m. The axis of the condenser array is vertical and aligned with the axis of the injection nozzle. This insures that the pumping gas will be directed toward the condensing unit and increases the probability of rapid condensation.

To use the inner structure in the cryoentrainment mode after the initial pumpdown, a remotely operated valve is used to completely isolate this structure from the pumping system. This isolation insures that any pumping phenomena observed in the inner structure is due solely to the entrainment mechanism and not the action of the action of the auxillary pumping system.

To initiate a typical operational cycle, the entire unit is evacuated to a pressure of near 10^{-6} torr prior to cooling the condensing unit. The initial pumpdown with a warm condenser insures that the condenser surface will not be contaiminated with large quantities of condensate (such as water vapor and carbon dioxide) prior to the operation as a cryoentrainment pump. Upon completion of the condenser cooldown with liquid nitrogen, helium is admitted to the system to provide the non-condensable test gas. To insure that the gas in the pump volume is essentially non-condensable, the initial base pressure is raised at least two orders of magnitude by the admission of helium. After isolating the inner structure at this new pressure, the entrainment fluid is admitted into the pump volume and the variation of the helium partial pressure observed as a function on time.

In order to monitor the helium partial pressure variation a partial pressure analyzer was mounted on the inner structure. This gauge is capable of sensing system total pressure to a maximum of 10^{-5} torr as well as partial pressures for species with mass numbers of 70 and below. To extend the total pressure range from 10^{-5} to 1 torr, a high pressure ionization gauge was used in conjunction with the partial pressure analy...

To operate the cryoentrainment system, two modes are possible with respect to the introduction of the pumping gas stream. The stream may be admitted either by using a continuous flow or by periodically injecting the fluid into the system in short bursts. The short burst method is favored over the continuous technique for several reasons. For a given pressure ratio between the stagnation chamber and pump volume, the gas stream will be accelerated to some exhaust velocity. In the continuous injection method, it is reasonable to assume that the pump pressure level will be slightly elevated as instantaneous trapping of all the injected mass will not occur. For the short burst method, on the other hand, it is to be expected that the background pressure will drop as condensation of the injected gas occurs between bursts. Thus, for the periodic burst technique, a higher pressure ratio should be available for accelerating the gas stream than for the continuous method.

Further disadvantages of the continuous method of operation are the consumption rate of the entrainment fluid and the rapid degrading of the condenser performance as a result of rapid condensate build-up.

For these reasons, the single burst approach was chosen as the method by which the entrainment fluid would be introduced. To accomplish this, a small volume, which may be charged to a known pressure with the pumping gas, was attached to the stagnation chamber. This volume is separated from the pump volume by a solenoid valve which is triggered by a timed circuit. In this way known quantities of the pumping gas are introducted into the system and with a reproduceability, for comparison of various experimental runs, not attainable by hand operation of the injection cycle.

For the examination of the ionization gauges at low temperatures, an existing system was employed. Basically, this system consists of two stainless steel volumes, connected by a capillary tube. Each of the volumes has provisions for the installation of a nude ionization gauge. Further, one of the volumes may be immersed in a liquid nitrogen bath while the other is maintained at room temperature.

Any anomalous behavior then noted on the gauge in the low temperature environment -- an abnormally low pressure indication, for example -- can then be correlated to the true conditions in the cooled tank by use of the gauge indication from the room temperature volume. This correlation may be made through the use of data previously acquired in this laboratory on thermal transpiration effects in long tubes. From these data, the conditions in the liquid nitrogen cooled volume may be determined from a knowledge of the conditions as measured in the room temperature volume.

DISCUSSION OF RESULTS

As the evaluation of the cryoentrainment pump proceeded, several areas in the operating technique that required improvement became apparent. In the early stages of the study, the condenser was filled until liquid nitrogen was flowing freely from the exit. At this point, the liquid nitrogen supply was cut off. Although implying that the condenser was initially full, this did not insure that it would remain so for a long period of time. Although liquid nitrogen was periodically introduced into the condenser, the resulting data suggested that the condenser temperature was not reaching a low value.

The data presented in Figure 2 are representative of those obtained with this technique using C_4F_8 as the pumping agent. An initial sharp fall in the helium partial pressure was observed as would be expected from the collisions of the pumping gas with the helium molecules. This drop in pressure -- from 1.5×10^{-5} torr to 1.1×10^{-6} torr is equivalent to a helium pumping speed of 780 l/sec.

The time period following the injection of the C_4F_8 is of critical interest in that the helium partial pressure rises quite rapidly to near its initial value. This suggests that only a minimal amount of pumping is occuring and that the condenser temperature was too high to permit rapid condensation. Further injections indicate, by the slightly increased retention of the helium that either the C_4F_8 is beginning to stick to the condensate present from previous injections or that the condenser temperature is being slightly lowered as the run progresses.

In an effort to lower the condenser surface temperature, modifications were made to the condenser vent line. A 50 cfm mechanical pump was attached to the vent line to lower the condenser ambient pressure, thereby lowering the condenser temperature. Due to the fact that the mechanical pump ingested liquid nitrogen when the condenser unit was completely full, this technique was not useable on a continuous basis. However, it was used periodically to insure that the condenser remained full and near a temperature of 77°K.

That the reducing of the condenser temperature was of critical importance is shown in Figure 3. In this instance, C_4F_8 was injected into the system at a stagnation chamber pressure of 380 torr, corresponding to an injected mass of 8.9×10^{-3} grams, after the inner structure was sealed at a helium partial pressure of 7.5×10^{-6} torr.

As noted in the figure, the partial pressure decreased to near its final value of 7.5×10^{-8} torr within approximately three seconds following the first injection of C_4F_8. Using the system volume of 900 liters and the simple exponential variation of pressure with time, this decrease corresponds to a helium pumping speed of 1500 l/sec.

The most important difference between this run and previous ones is the variation of the helium partial pressure with time after the injection of the C_4F_8 gas. As noted in Figure 3, the cryodeposit is highly effective in retaining the trapped helium.

Following the first injection of C_4F_8, the system is apparently establishing some new equilibrium condition. A slow rise in the helium partial pressure is initially noted which corresponds to a desorption rate of 7.5×10^{-8} torr liter/sec. For the time period beyond this initial adjustment phase, the change in the helium partial pressure with time was not great enough to be measured.

The initial partial pressure rise is due to the desorption of helium molecules bound loosely on or near the surface of the cryodeposit. Although not indicated in these data, the trapped helium is routinely held in the cryodeposit for times in excess of one hour following a single burst of C_4F_8. Desorption rates for these longer times are typically on the order of 1.5×10^{-8} torr liters/sec.

Further injections of C_4F_8 did not appreciably alter the minimum value of the helium partial pressure as is seen in Figure 3. The initial burst of C_4F_8 removed 99 percent of the helium molecules initially present in the system. Thus an examination of the change in the probability of collision following the first injection indicates that the helium removal rate must be lowered significantly.

When a gas stream is passed through a stationary target gas, the probability of collisions between the two types of molecules is inversely proportional to the mean free path of the target gas. In this instance, the helium mean free path length has been increased by a factor of 100 following the first injection. Therefore, the collision probability for the C_4F_8 helium is decreased by a factor of 100 for the second injection. As a result the rate of helium removal is sharply reduced.

A further factor influencing the helium removal rate is the condensation process of the injected gas stream. After an injection, 100 percent of the injected gas is not immediately condensed. Thus, for the second injection of the pumping gas, the entrainment effect is shared between the helium molecules and those of the pumping gas not yet condensed from the prior injection.

One other phenomena that might be of use in reducing the base pressure for a system such as this is that of crysorption. However, as indicated by the constant or slowly rising value of the helium partial pressure, no measureable sorption pumping by the cryodeposit is occuring at this temperature and pressure. This is in agreement with trends shown by isotherms for various cryodeposits in that the sorption capacity falls rapidly as the cryodeposit temperature is raised above 20°K.

Carbon dioxide was also employed in the system to examine the effects of a lower molecular weight molecule. The results of a typical cycle are shown in Figure 4.

The time variation of the helium partial pressure following an injection of carbon dioxide is qualitatively the same as that for C_4F_8. The decrease from the initial helium partial pressure of 4×10^{-6} torr is equivivalent to a helium pumping speed of 830 l/sec using carbon dioxide as compared to the 1500 l/sec speed obtained with C_4F_8.

The helium partial pressure is further reduced by additional injections of carbon dioxide although the pumping speed is sharply reduced after the first injection. For the second injection, the pumping speed is 8.75 l/sec and is further decreased to 6.6 l/sec for the third injection. This abrupt decrease in pumping speed is attributable to the effects of both the decreasing collision probability with the helium as well as the increased number of collisions with like molecules for the second and third injection of carbon dixoide.

The pumping speed of the cryoentrainment pump is a function of the injection pressure as well as the number of prior injections as indicated by the previous data. To obtain a preliminary estimate of the magnitude of these effects, an injection of C_4F_8 at 250 torr was made into an initially pure helium environment. The pumping speed obtained for this condition was 52.2 l/sec. This value indicates the significance of the injection pressure when compared to the 1500 l/sec speed obtained for a 380 torr initial injection as in the data of Figure 3. Following the 280 torr injection pressure was increased to 380 torr to examine the influence of free entrainment fluid molecules that had not been condensed after the first injection. The pumping speed for this injection condition was found to be 810 l/sec. Then the sensitivity of the system to the number of prior injections may easily be seen by again noting that the pump speed for a 380 torr injection into an initially pure helium environment is 1500 l/sec.

Since it is desirable to condense as much of the injected gas as possible, both to allow continued high pumping speeds and to permit low background pressures to be reached, examinations of the total pressure variation after an injection of pumping gas were made.

The variation of the system total pressure following several injections of carbon dioxide is shown in Figure 5. It is apparent that the system pressure is increased with each injection; a characteristic not desirable in a vacuum pump. The pressure data for the carbon dioxide injections indicate that the surface temperature is near 105°K rather than the anticipated value of 77°K. Since the conditions in the vacuum environment are below the triple point for carbon dioxide, it is felt that a porous deposit is formed on the condenser rather than a dense ice. As a result, the heat transfer characteristics of the cryodeposit are poor, allowing a high surface temperature.

The system total pressure variation following an injection of C_4F_8 is presented in Figure 6. A behavior similar to that for carbon dioxide is immediately apparent. Although vapor pressure -- temperature data were not available for C_4F_8 it was assumed that a deposit similar to that of carbon dioxide was formed with a resultant high surface temperature.

To further investigate the characteristics of the various cryodeposits, a second small system was constructed. Viewing ports were installed so that visual observation of the deposit formed on 77°K stainless steel tubes was possible. After bleeding in C_4F_8 to establish a deposit, it was found that portions of the frost could be dislodged by vibration and that the frost did in fact appear to be highly porous. Thus the deposit formed in the cryoentrainment pump did have a high surface temperature with a correspondingly high vapor pressure. A further aid in establishing a high background pressure, as in Figure 6, was the flaking off of the condensate under the action of vibration and re-entering the system through evaporation.

Mass spectra obtained during the C_4F_8 pumping cycles indicated that the common gases, such as water vapor, were evacuated to below measureable levels by the entrainment and trapping mechanism. The remaining components were of mass numbers 16 and 31. A mass number of 31 corresponds to CF, the major cracking fraction of C_4F_8 present in the range of the instrumentation used. The mass number 16 component, which is introduced with the injection of C_4F_8 gas, is perhaps due to the imperfect replacement of hydrogen of fluorine in the production of C_4F_8. This would then yield constituents such as CH_4.

PLAN FOR FUTURE WORK

To improve the efficiency of the cryoentrainment pump more rapid condensation of the injected gas and a lower condensate surface temperature need to be achieved. To accomplish this, the condenser will be redesigned to provide a larger surface area than that now available and the temperature of the refrigerant will be lowered.

To attain a larger area for the condenser, the new installation will take the form of an array of finned tubes with the fins parallel to the jet axis. These units will be stacked one under the other to provide for multiple collisions of the molecules with cold walls while increasing the available area. A second baffle will also be positioned below the condenser to prevent any condensate that falls from the unit from re-entering the system by evaporation. Preliminary estimates indicate that the present surface area should be increased by a factor of six to limit the temperature rise through the frost to $5^{\circ}K$.

As an aid to lowering the surface temperature, the baffle system leading to the vacuum pump used for sub-cooling will be modified so that it may be used continuously rather than intermittantly as is the situation presently. Using this technique and maintaining a pressure of one-half an atmosphere above the liquid nitrogen should yield a temperature of $69^{\circ}K$. The combination of greater surface area and lower refrigerant temperature should then give conditions near those required for a carbon dioxide vapor pressure of 10^{-10} torr.

After installation of the new condenser, studies will be made to again determine the optimum operational technique for the system.

A secondary investigation will also be performed with the small system incorporating the viewing ports. A study will be made of the ability of gases, such as C_4F_8 and carbon dioxide, to adhere to different metals at 77°K. An analysis will also be made of various surface treatments of the metals used, in an attempt to provide better surface adhesion.

At the present time no meaningful data has been generated in the low temperature ionization gauge study. This is due to the unreliability of the system as caused by the repair downtime of the ionization gauges as well as loss of system integrity from numerous and repeated thermal shocks when cooling to 77°K. Repairs of the system elements have been completed with the system joints now formed solely by fusion welding or metal gasketed joints which are highly resistant to thermal shock. This system may now be used confidently to examine the effects of low temperature on ionization gauges. Further studies in this area will be carried out jointly with that of the cryoentrainment pump under the continuation grant NASA NGR No. 34-002-106.

CONCLUSION

The cryoentrainment pump promises to be an effective system for providing a clean vacuum both rapidly and economically. Pumping fluid consumption is low with 8.9 x 10^{-3} grams providing a pumping speed of 1500 l/sec and a retention time in the cryodeposit in excess of one hour.

The use of the cryodeposit provides a means of removing non-condensable spacies from the system of moderate cryogenic temperatures without the use of secondary devices such as oil diffusion pumps.

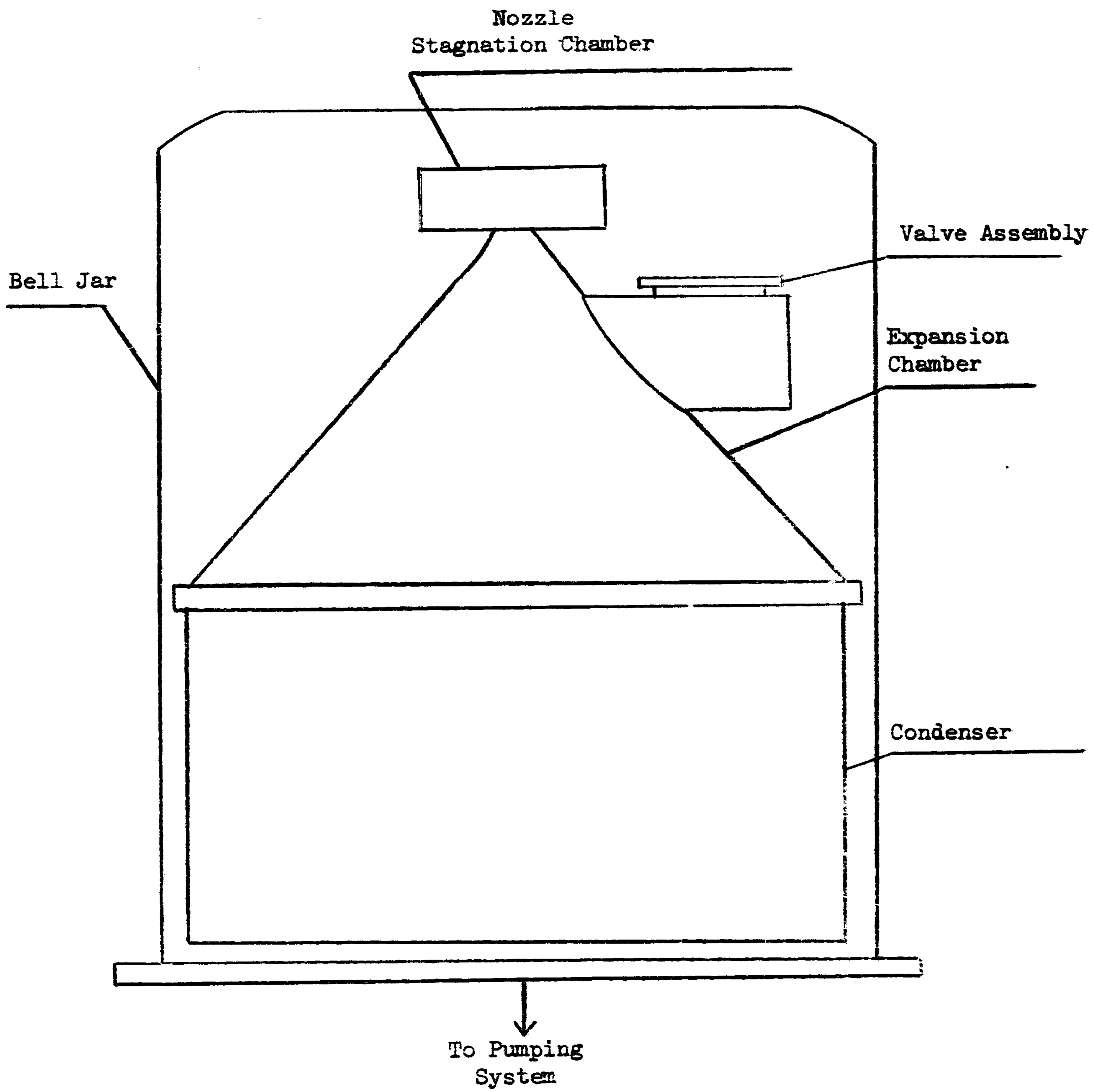

Figure 1. Schematic of the Experimental Apparatus

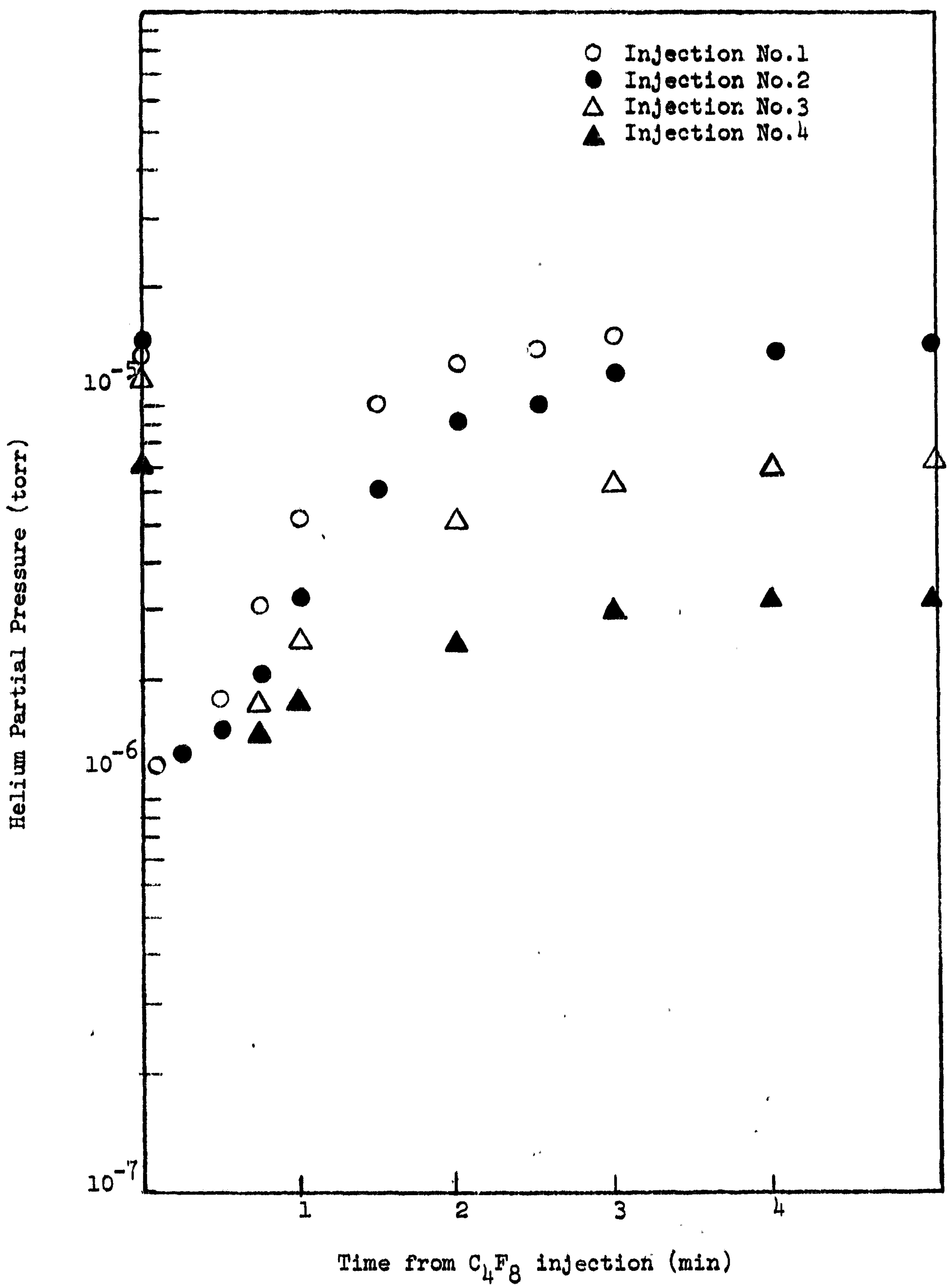

Figure 2. Helium pumping by C_4F_8 injection

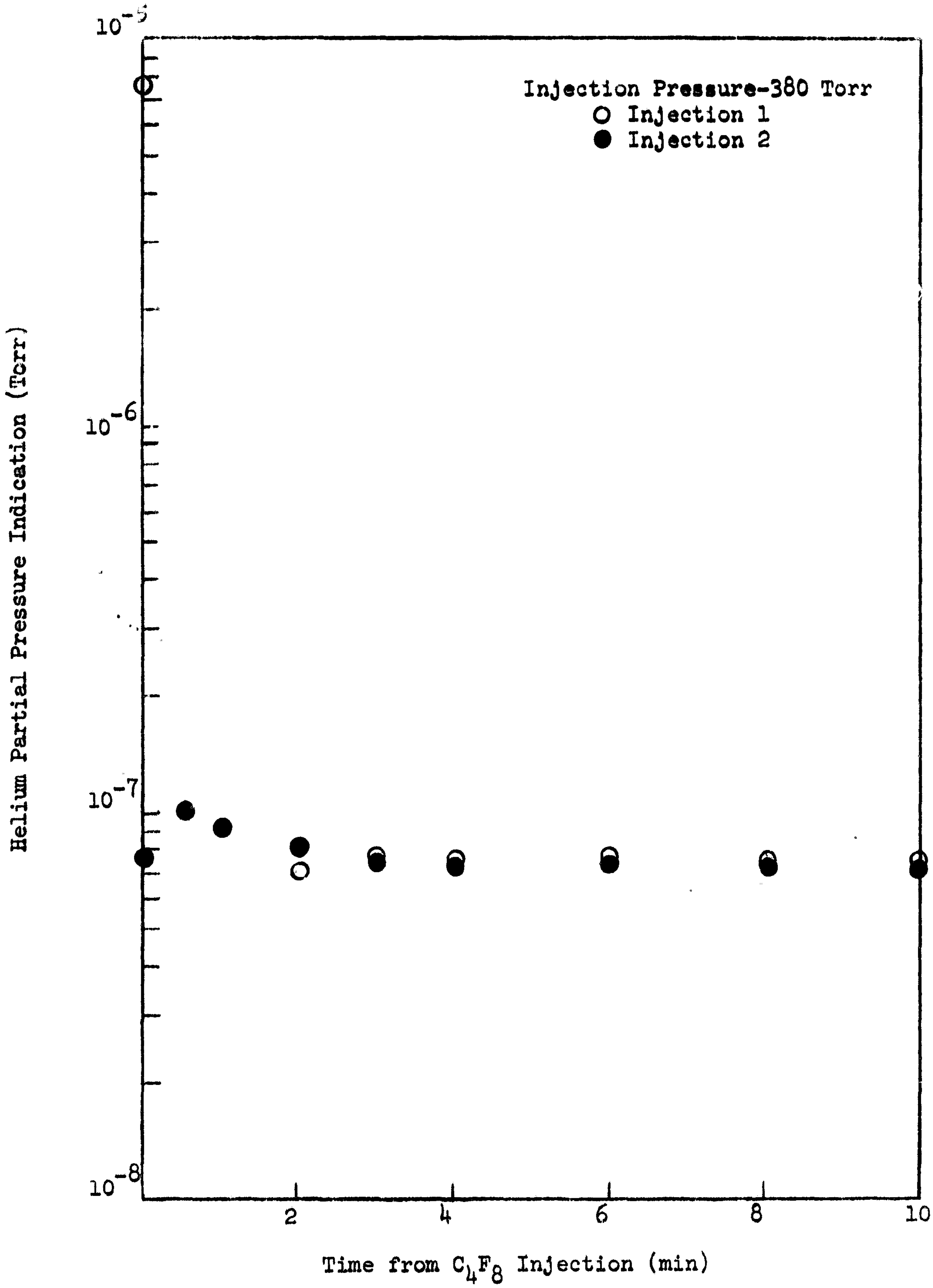

Figure 3. Hel um Entrainment by C_4F_8

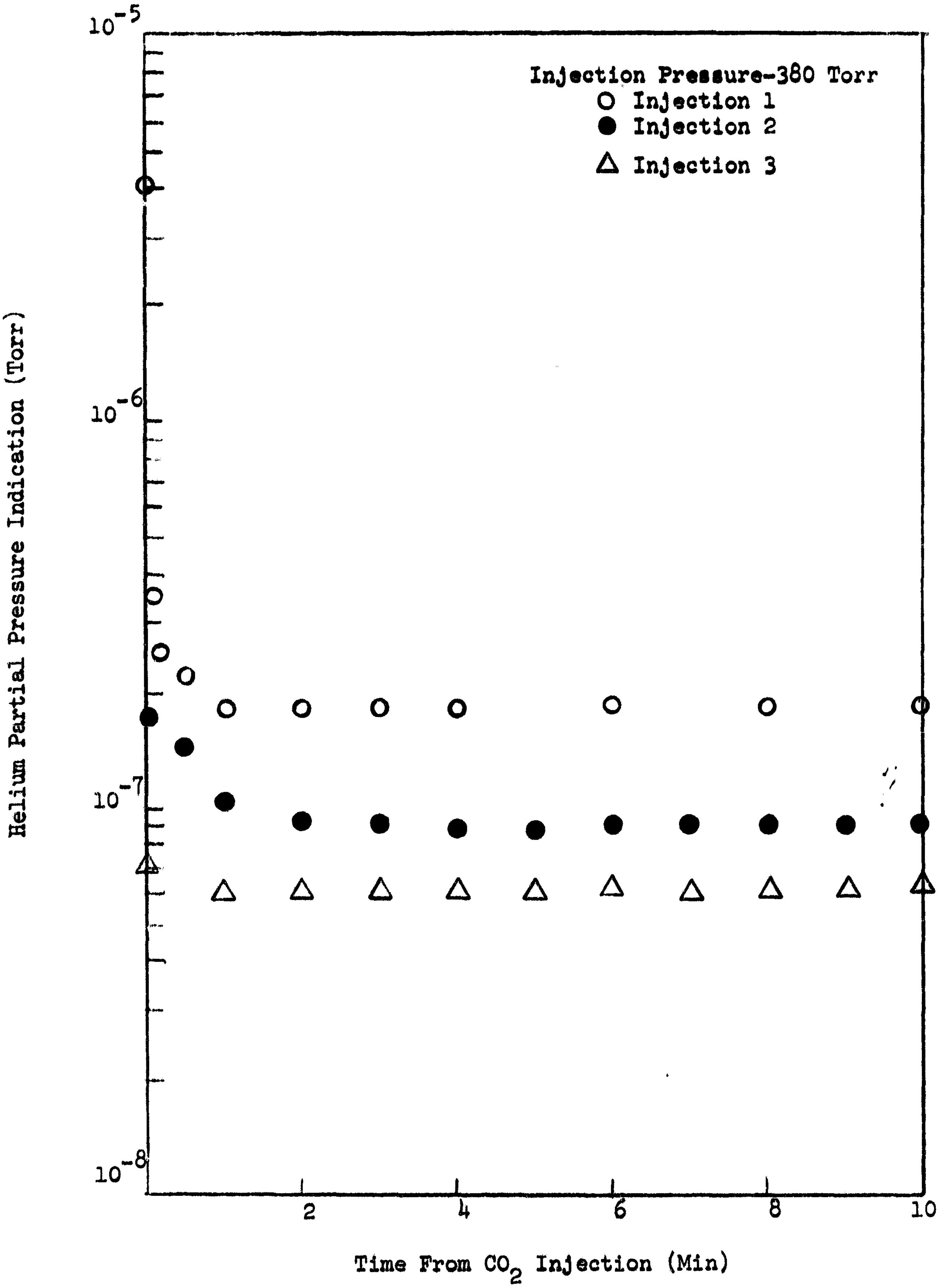

Figure 4. Helium Entrainment by CO_2

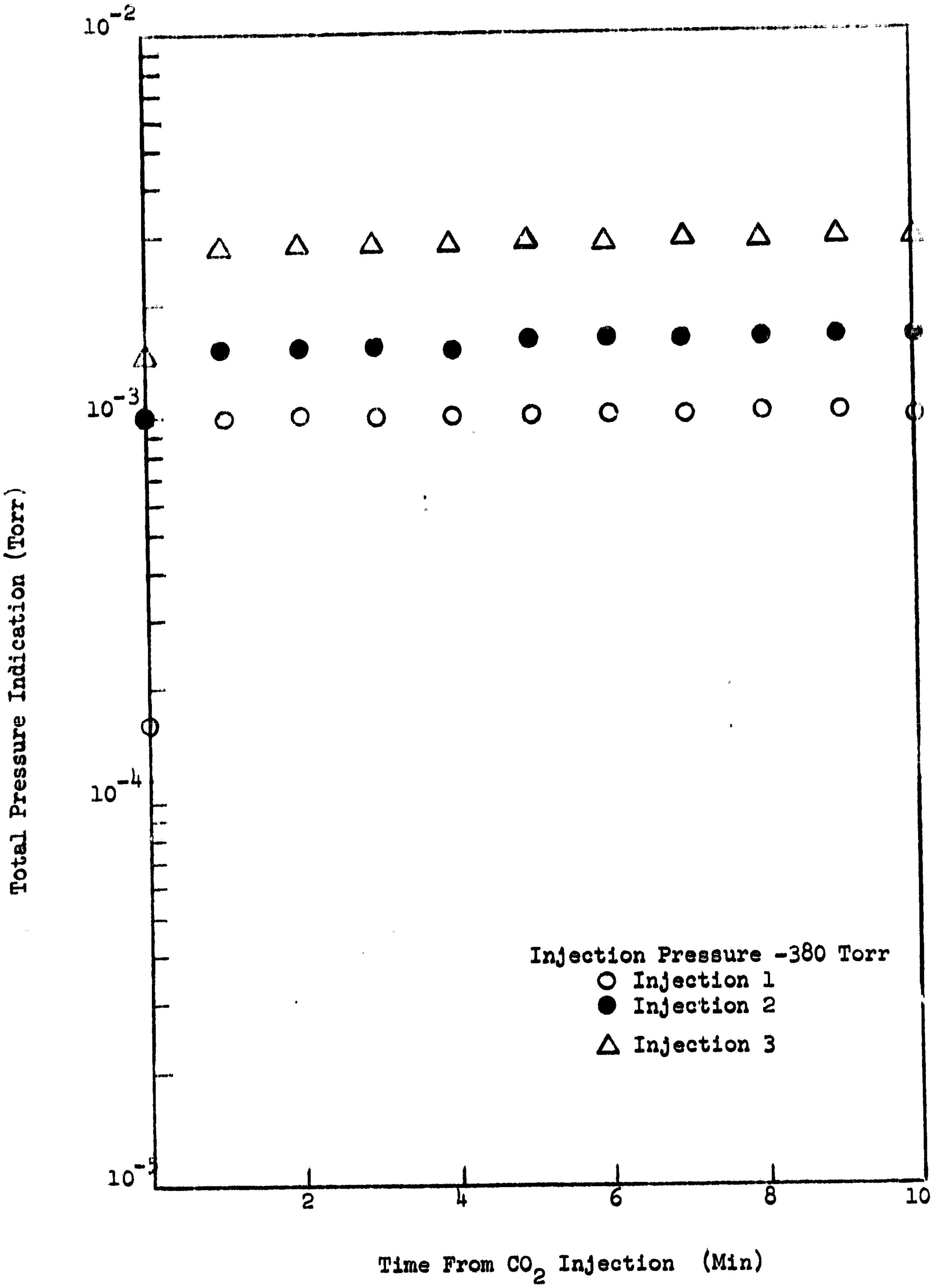

Figure 5. Total Pressure Variation with CO_2 Injection